Ionization chamber for high-temperature gas chromatography

Simultaneous monitoring of the effluent stream from gas chromatography equipment containing radioactive compounds by an ion chamber in series with the usual mass-detecting thermal-conductivity cell has been described by WILZBACH AND RIESZ[1]. However, when this design of ion chamber employing a ceramic insulator was used at 200° to 250°, as required for the gas chromatography of fatty acid esters, strain currents limited its applicability.

Construction of an ionization chamber from readily available materials is described which is applicable to high-temperature (240°) gas chromatography.

A successful model, illustrated in Fig. 1, based upon the use of Teflon, was evolved after studying a number of potentially useful high dielectric constant materials. Teflon comprises the main insulator *a*, which is threaded to receive the brass can *l*, support plugs *b* and *c*, and inlet and outlet connectors *d* and *e*. The ion chamber *l* has an internal working volume of about 9 ml.

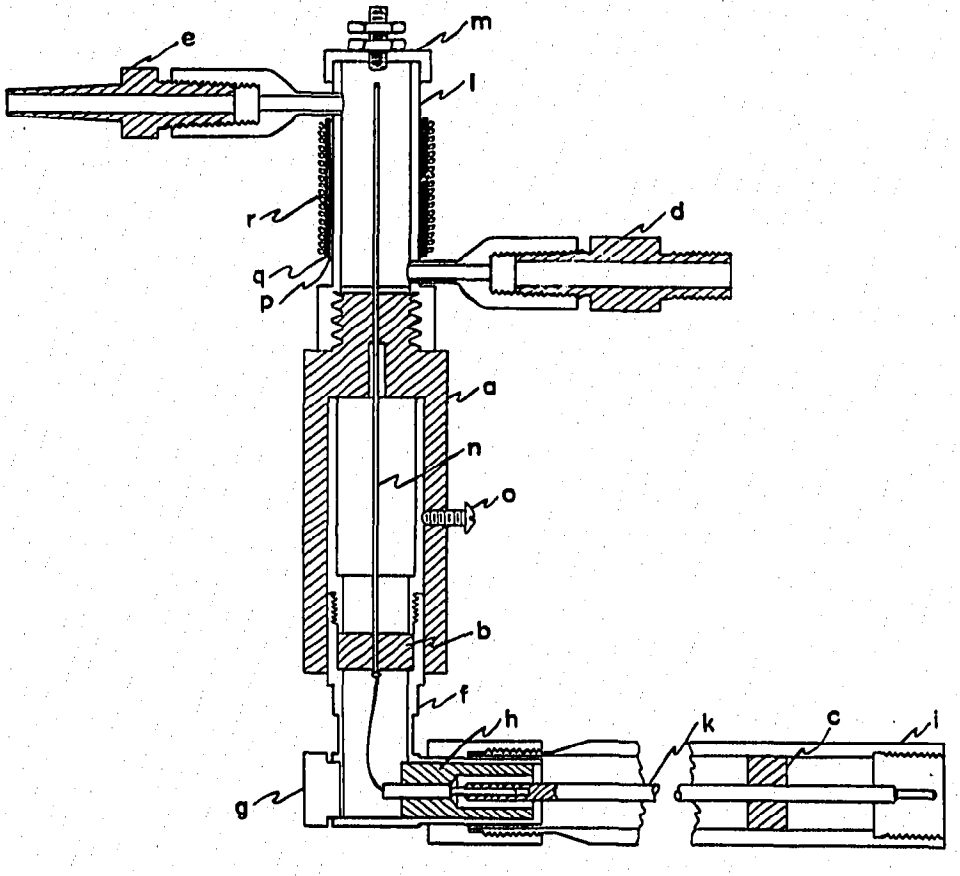

Fig. 1. Ionization chamber.

The probe *n* is constructed from BSA–18 gauge tungsten rod and is connected to the brass contact pin through a short piece of copper wire silver-soldered to the probe and soft-soldered to the contact pin. A hole is drilled in the top of an Amphenol 82–833 fitting *f* and closed with brass plug *g*. Internal parts of the fitting, except the polystyrene insulator *h* and its brass contact pin, are removed. The modified fitting is connected to the preamplifier head of a Brown Electrometer through the coaxial "cable" consisting of a threaded brass tube *i*, a brass rod *k*, and plug *c*. A current of air is used to cool the tube to prevent heat transfer from the chamber to the preamplifier. Brass cap *m* is provided with a bolt and nuts for connection to the negative side of a 4.5-volt battery. The positive side is grounded on the preamplifier case.

Around the ion chamber *l* is wrapped a layer of asbestos *p*, a grounded metal shield *q*, and a layer of No. 26 asbestos-covered nichrome wire *r*. Movement of insulator *a* relative to a brass shield, which screws into the coaxial fitting, is prevented by set screw *o*.

In operation the chamber is padded with Pyrex wool and covered with a grounded metal shield containing the necessary exit holes for gas streams and electrical leads. This shield eliminates capacity effects and lessens heat loss. Pyrex wool and asbestos paper are used to insulate the standard taper inlet connector *e* after it is plugged into the exit connector from the thermal conductivity gauge of the chromatography equipment, *i.e.*, Aerograph. Current is supplied to the heater wiring with a variable autotransformer. The temperature is adjusted by temporary insertion of a thermometer in place of connector *d*.

Fig. 2 illustrates results obtained with about 2 mg (3 μC) of tritium labeled methyl stearate[2] using a 5-foot Resoflex 296 column[3] operated at 205° and at a

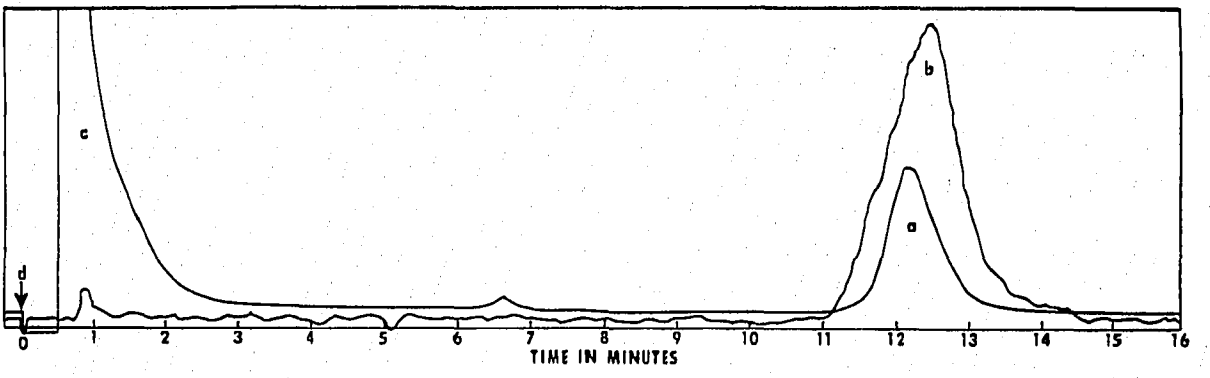

Fig. 2. Gas chromatography of tritiated methyl stearate. a. Thermal conductivity peak. b. Radioactivity (ion current) peak. c. Solvent peak. d. Point of injection.

helium flow rate of 44 ml/min. The thermal conductivity data are recorded at 2 mV, full-scale sensitivity, and ion currents are recorded using the 10 mV range of the electrometer. Both recorder charts were run at 60 inches per minute and are superimposed in Fig. 2. At the specific flow rate used in this equipment, radioactivity lags thermal conductivity by about 20 seconds. These curves demonstrate the chromatographic identity of tritium labeled stearate with inactive stearate and indicate a small amount of labile tritium contaminant.

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[1] K. E. WILZBACH AND P. RIESZ, *Science*, 126 (1957) 748.
[2] H. J. DUTTON, E. P. JONES, L. H. MASON AND R. F. NYSTROM, *Chem. and Ind. (London)*, (1958) 1176.
[3] S. R. LIPSKY AND R. A. LANDOWNE, *Conference on the Analysis of Mixtures of Volatile Substances*, New York Academy of Sciences, New York, April 10, 1958.

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